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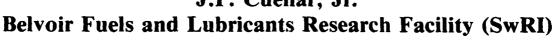
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DEVELOPMENT OF A TEST METHOD TO DETERMINE POTENTIAL PEROXIDE CONTENT IN TURBINE FUELS

INTERIM REPORT BFLRF No. 199

Ву

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Southwest Research Institute San Antonio, Texas

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			sought so that				
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			storage method ls' oxidative to				
_			allow selection				
							ts. Additionally,
							reaction kinetics
to aid	the deter	rmination of	fuel peroxidation	on potential	. From a pa	artial	lly completed
experi	mental mat	trix, fuel st	fuel peroxidation ressing was comp	oleted at 60	and 100°C	undei	r an initial
oxygen	pressure	of 689 kPa (100 psig). Expe	erimental re	sults at 60°	'C gav	ve inconclusive
result:	s. Result	s of the 100	°C experiments ;	produced ess	entially sel	lf-cor	nsistent results
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19.	ABSTRACT:
	that also agreed with those of the bottle storage for the most stable and least stable fuels. Results of the two intermediate stability fuels, however, were interchanged.

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FOREWORD/ACKNOWLEDGEMENTS

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INTRODUCTION

Background

In 1976 the U.S. Navy experienced difficulties with A-7E aircraft that operated in the western Pacific. It was shown^{(1)*} that these difficulties were caused by peroxides in the turbine fuel that attacked the neoprene diaphram in the engine's fuel pump. To avoid similar problems in the future, it would be advantageous to have a test method that would predict the maximum, or potential, peroxide content of a fuel even before the fuel has any measurable peroxide content. Thus far, attempts to predict fuel peroxidation tendencies were either too time consuming or produced false trends relative to ambient temperature results.

Objective

The objective of this study was to develop a practical test method for the prediction of the peroxide potential of fuels.

Approach

A study was made of the effects of oxygen concentration (pressure), temperature, and test duration on selected kerosene-type fuels. As indicators of the extent of fuel oxidation, oxygen uptake, peroxide content, formation of gum, water, and acidity were measured. These data were then compared to the results of a generally accepted long-term "bottle storage test" conducted at 43°C.

Superscript numbers in parentheses indicate references listed at the end of this report.

II. TEST FUELS

Four kerosenes were selected to serve as "model" fuels for the initial phases of the program. One of these fuels (No. 0464) was a straight-run, salt-dried, and claytreated, additive-free Jet A, to serve as a pristine, stable fuel. Two hydrocracked kerosenes were selected as potentially unstable fuels. Each of these unstable fuels was also percolated through a column of activated alumina to remove the peroxides and other polar components. Since one of the hydrocracked kerosenes (No. 11310) contained high concentrations of peroxides (420 ppm), it was only used after the alumina treatment. The other hydrocracked fuel (No. 11381) was used in the oxidation experiments both before and after the alumina treatment.

Chemical reactivity, including oxidizability of a fuel, depends upon the fuel's chemical composition. Thus, in addition to the ASTM-type analyses summarized in Table 1, a hydrocarbon-type analysis was also performed on each of the model fuels by ¹H and ¹³C nuclear magnetic resonance (NMR) spectroscopy. Proton-type assignments were made according to the recommendations by Netzel and Hunter. (2) Abbreviated NMR definitions are given in Table 2, while the spectroscopic data are summarized in Table 3.

It should be noted that the $H(\alpha-1)$ protons are reactive benzylic types, and that $H(\beta-N)$ include naphthene hydrogens beta to an aromatic ring and hydrogens on tertiary carbon atoms. It was expected that fuels with widely different oxidative tendencies would exhibit gross differences in their NMR spectra, especially in their $H(\alpha-1)$ and $H(\beta-N)$ regions. Thus far, this expectation has not been fulfilled. The concentrations of reactive sites in Table 3 as measured by NMR do not reflect the relatively large differences in the reactivity of the test fuels with oxygen.

TABLE 1. ANALYSIS OF SELECTED KEROSENES

Property	ASTM Designation	0464(*)	Fuel Number 11310(**)	11381(**)	JP-5 Spec (partial) MIL-T-5624-L
Specific Gravity @ 15.50C	D 1298	0.8185	0.7976	0.8174	0.788 - 0.845
Flash Point, OC	D 93	45	47	99	60 min
Freeze Point, OC	D 2386	-41	-44	-40	-46 max
Smoke Point, mm		19.2	24.7	20.8	19.0 min
Sulfur, wt%	D 2622	0.03	0.01	0.01	
Hydrocarbon Types, vol%	D 1319				
Saturates		80.7	87.8	78.8	
Olefins		1.4	0.5	0.7	5.0 max
Aromatics		17.9	11.7	20.5	25.0 max
Distillation	D 86				
Initial Boiling Point, OC		150	150	181	
10% Recovered, OC		182	166	198	205 max
50% Recovered, OC		219	192	219	
90% Recovered, OC		256	244	253	
End Point, OC		284	281	273	290 max
Heat of Combustion,					
Gross, Btu/lb	D 240	19,691	20,134	19,700	
Heat of Combustion,					
Net, Btu/lb	D 240	18,437	18,851	18,442	18,300 min
Oxidation Stability, 16 hr	D 873				
Soluble gum, mg/100 mL		3.4	90.5	6.8	
Insoluble gum, mg/100 mL		0.0	0.0	0.4	
Precipitate, mg/100 mL		0.0	0.7	0.7	
Total potential residue		3.4	92.1	7.9	
Peroxide Number, ppm	D 3703	0.0	420	2.8	

Straight-run, salt-dried, and clay-treated additive-free Jet A Hydrocracked kerosene

TABLE 2. PROTON NMR DEFINITIONS

Symbol		Definitions	Chemical Shift Range (ppm from TMS)
H(AR)	=	Total Aromatic Hydrogens	6.6-8.3
H(OL)	=	Olefinic Hydrogens	4.0-6.0
H(α−1)	=	Methylene Hydrogens Alpha to Aromatic Ring	g 2.3-4.0
H(α−2)	=	Methyl Hydrogens Alpha to Aromatic Ring	1.9-2.3
H(β-N)	=	Naphthene Hydrogens Beta to Aromatic Ring and Hydrogen on Tertiary Carbon Atoms	1.6-1.9
Н(β)	=	$^{\beta}\text{-CH}_2\text{-and}$ $^{\beta}$ -CH $_3$ to Aromatic Ring and Normal Alkane -CH $_2\text{-Hydrogens}$	1.0-1.6
H(Y)	=	Y-CH3 to Aromatic Ring and Normal or Branch Alkane -CH3 Hydrogens	0.5-1.0

Source: Reference 2.

TABLE 3. HYDROGEN-TYPE ANALYSIS BY PROTON NMR SPECTROSCOPY

Percent Hydrogen Type in Fuels 0464 Н Туре 11381 11381/Al₂O₃ 11310/Al₂O₃ H(AR) 3.0 2.9 2.7 2.4 0.0 0.0 0.0 H(OL) 0.0 3.4 3.2 3.2 H(α -1) 1.0 4.3 4.8 4.3 H(α -2) 1.9 H(3-N) 4.8 6.2 5.3 4.8 H(3) 50.5 50.2 48.9 50.1 32.8 33.8 35.5 H(Y) 39.8

IIL APPARATUS AND PROCEDURES

To establish baseline data on the long-term stability of the four selected "model" fuels, the generally accepted method of bottle storage at $43^{\circ}C^{(3)}$ was used. In this procedure, 200 mL of each of the test fuels were saturated with "synthetic" air (21% O₂ and 79% N₂, purified) at 300 mL per minute for 5 minutes. Then the fuel samples were stored for an extended time in the dark at $43^{\circ}C$ in sealed 500-mL amber borosilicate bottles. After aging periods of 1, 2, 3, and 4 weeks, followed by 4-week intervals, one bottle of each fuel was retrieved for analysis. The oxygen content was determined by GC in both the liquid and vapor phases, in addition to measurement of peroxides, gum, water, and acid number of the liquid phase. If the oxygen concentration in the vapor phase was below 10.0 vol%, the remaining bottles of the same fuel were again aerated, as described above.

Experimental accelerated oxidative stressing of these fuels was done in a pressure reactor. The reactor was made of Type 316 stainless steel equipped with a magnetically-coupled stirrer driven by a variable speed motor, an external electrical heating element, and an internal cooling loop with automatic temperature control. Valves and fittings permit introduction of gases and liquids and withdrawal of samples either at the bottom or the top of the vessel. The sample is held in a borosilicate reactor liner. Additionally, the reactor is equipped with a 1380 kPa (200 psig) rupture disc, and a 1380 kPa (200 psig) recording pressure gauge, both of which are replaceable with items rated at pressures up to 13,790 kPa (2000 psig). In laboratory practice, the borosilicate reactor liner is charged with 300 mL of fuel. The assembled system is then purged with oxygen of ultra-high purity (99.99% min), followed by pressurization to a predetermined pressure, e.g., 689 kPa (100 psig). While the fuel is stirred at about 150 rpm, the fuel temperature is raised to the test temperature within 20-30 minutes, and held there for the test duration within a tolerance of +0.5°C. The pressure and temperature within the reactor are continuously recorded to allow the calculation of the amount of oxygen that reacted with the fuel. Upon completion of the experiment, the reactor is disassembled at room temperature and atmospheric pressure. All the interior parts are washed, in several increments, with a total of 30 mL of 2-propanol to collect and dissolve all the formed water. The formed gum is dissolved in minimum measured amounts of an equivolume mixture of toluene, acetone, and methanol (TAM). These washings are added to the fuel that is analyzed for peroxides (ASTM D 3703), gum (ASTM D 381), water (ASTM D 1744), and acid number (ASTM D 3242).

IV. RESULTS AND DISCUSSION

Bottle storage of fuels under atomospheric air at 43°C established the ranking of the four fuels in order of their expected storage stability. The experimental data are summarized in Table 4. The same data are graphically presented in Figures 1 through 5, in which time dependence is illustrated for oxygen consumption in the vapor and liquid phases, as well as that of the generation of peroxides, water, and gum, respectively. Within experimental error, each of the variables give the same ranking of fuels' storage stability, that is, the order of decreasing stability is: Fuel Nos. 0464, 11381, alumina-treated 11310, and alumina-treated 11381.

It is a generally accepted approximation that storage for about 12 weeks at 43°C has about the same deteriorating effect on a fuel as does a full year of storage under ambient temperature conditions. Since such a lengthy procedure is not suitable for quality control, the goal of this project was to find accelerated fuel-aging conditions that yield comparable results. It was a further goal to design the experimental matrix in such a way that the results would be usable for a chemical kinetics treatment. In the experimental matrix, a cross-correlation was sought among time, temperature, and oxygen pressure effect as summarized:

Temperature, OC	Pr	essure,	osig
60	50	100	150
80	50	100	150
100	50	100	150

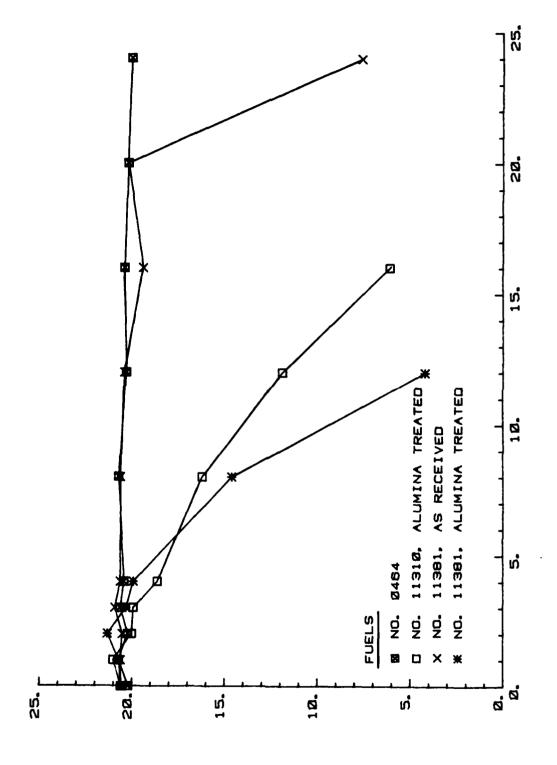
In this report, experimental results of fuel oxidations are described under the influence of 689 kPa (100 psig) oxygen at 60° and 100°C for stress durations up to 48 hours, as summarized in Tables 5 and 6, respectively. Corresponding graphical illustrations of data are given in Figures 6 through 9 and 10 through 13, respectively.

Data at 60°C were obtained only after 24- and 48-hour stress periods. Oxygen depletion during the 60°C experiments, as measured by the pressure gauge, was insignificant. Oxygen uptake, as calculated from pressure gauge readings at room temperature before and after the experiments, leveled off at a low value of about 19.2 mmole/L of fuel (with a standard deviation of 1.8). The peroxide buildup paralleled the results obtained during the bottle storage. However, the quantities of

TABLE 4. BOTTLE STORAGE OF KEROSENES UNDER AIR AT 43°C

Fuel No.	Storage, weeks 0 1 2	0 ₂ in Vapor Phase, vol % 20.2	0 ₂ ,	Peroxides,	S.J. Gum, mg/100 mL	H ₂ O,
	0	Phase, vol %	_	ppm	mg/100 mL	
464	1					ppm
	1		68	0	0.2	
		20.7	61	0	3.8	
		20.2	62	0	0.0	
	3	20.6	61	0	1.9	
	4	20.4	58	0	0.5	
	8	20.7	64	0	0.3	
	12	20.3	57	2	1.3	59
	16	20.4	45	2	1.2	41
	20	20.2	59	2	0.7	36
	24	20.0	61	2	0.9	51
11310/A1 ₂ 0 ₃	0	20.6	70	0	0.6	
2 3	1	21.0	55	5	3.4	
	2	20.0	71	14	0.2	
	3	19.9	62	26	0.8	
	4	18.6	57	54	0.5	
	8	16.2	53	124	0.5	
	12	11.9	33	243	0.4	67
	16*	6.1	0	380	1.0	62
	20	13.6	43	599	0.6	74
	24	4.8	5	841	0.6	159
11381 "As Is"	0	20.6	72	1	2.6	
	1	20.6	59	3	5.9	
	2 3	20.5	60	2	1.6	
		20.9	58	2	1.7	
	4	20.6	59	4	0.9	
	8	20.6	61	8	0.4	
	12	20.4	60	5	1.2	43
	16	19.4	41	34	1.7	32
	20	20.2	59	16	0.8	59
	24	7.6	5	362	2.0	59
11381/A1 ₂ 0 ₃	0	20.6	69	0	1.0	
2 J	1	20.7	54	2	4.4	
	2	21.3	63	6	0.8	
	1 2 3 4	20.3	59	11	1.1	
	4	19.9	50	21	0.4	
	8	14.6	42	137	0.3	
	12*	4.2	0	386	1.4	72
	16*	8.2	0	610	2.6	79
	20*	4.3	6	1119	2.7	167
	24	4.8	0	1471	6.1	258

^{*}Reaerated

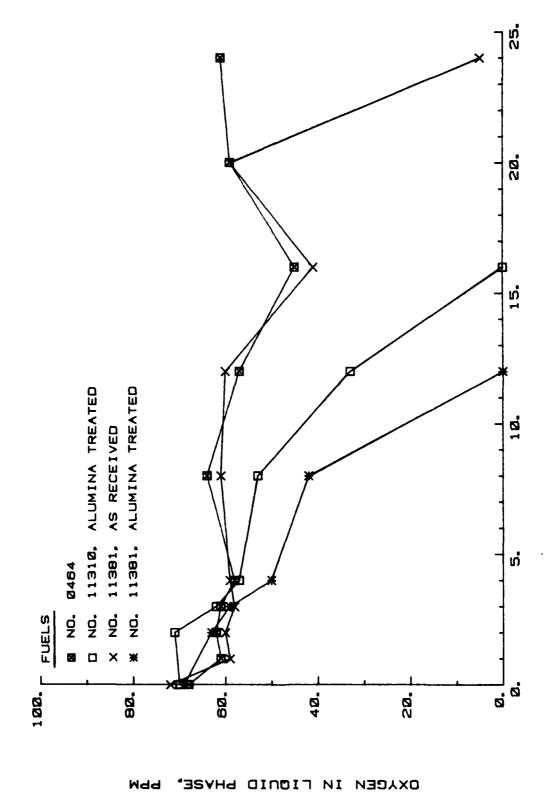


PIGURE 1. BOTTLE STORAGE UNDER AIR AT 430C (OXYGEN IN VAPOR PHASE, PERCENT)

STORAGE TIME, WEEKS

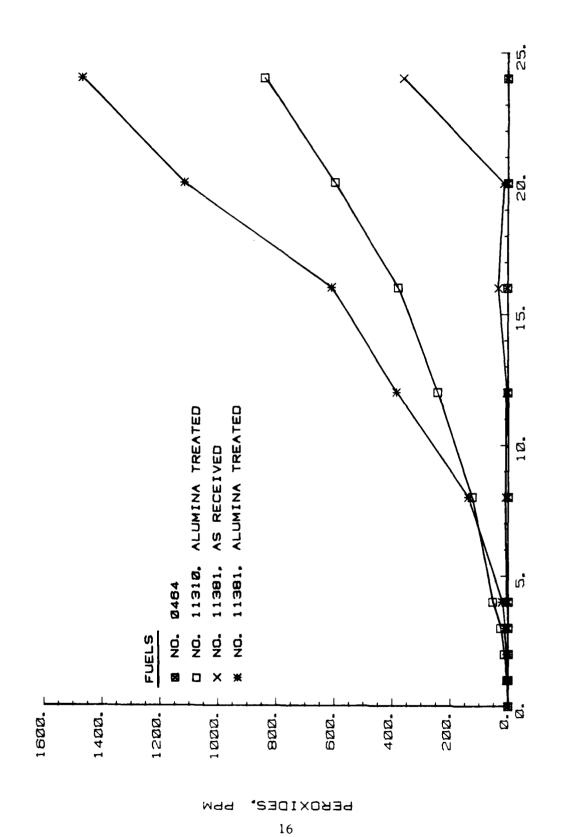
OXACEN IN AVEOR PHASE,

PERCENT



PIGURE 2. BOTTLE STORAGE UNDER AIR AT 43°C (OXYGEN IN LIQUID PHASE, PPM)

STORAGE TIME, WEEKS



PIGURE 3. BOTTLE STORAGE UNDER AIR AT 43°C (PEROXIDES, PPM)

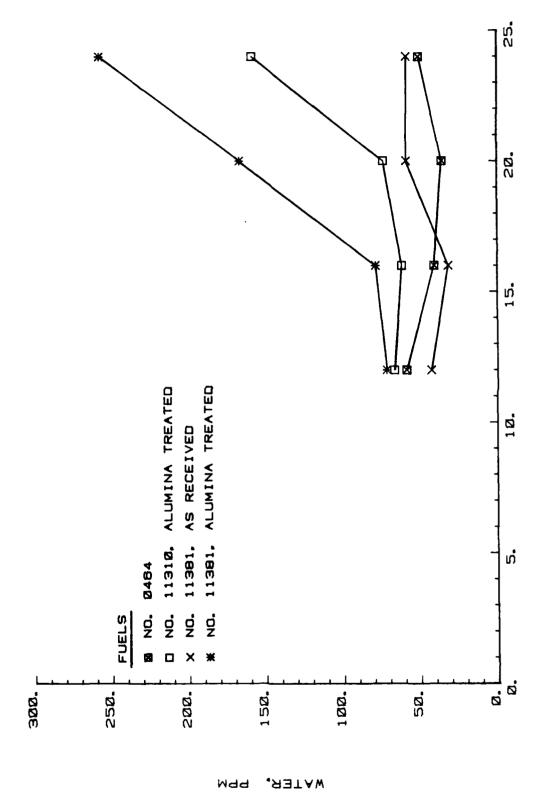
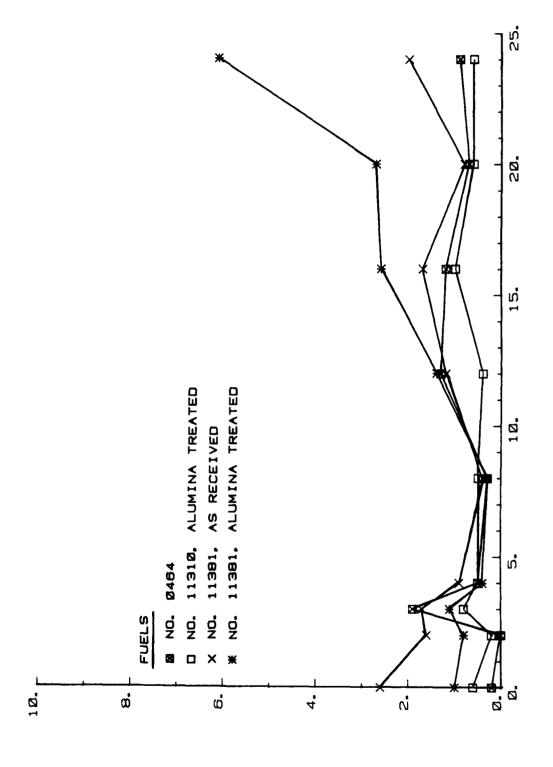


FIGURE 4. BOTTLE STORAGE UNDER AIR AT 43°C (WATER, PPM)

STORAGE TIME, WEEKS



PIGURE 5. BOTTLE STORAGE UNDER AIR AT 43°C (GUM, MG/100 ML)

STORAGE TIME, WEEKS

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MC

TW ØØI

TABLE 5. OXIDATION OF KEROSENES AT 60°C UNDER AN INITIAL OXYGEN PRESSURE OF 100 PSIC

		Time,	Oxygen Used,	Peroxides	5.3.Cum,	Water,	TAN,				Š	yken	Deple	Oxygen Depletion (psi) Vs Time (bours)	ps t	S S	() ()	urs)		
Fuel No.	Dat e	ă	monole/L	udd	mg/100mL	wdd	mg KOH/B	-	2	7	80	21	16 2	20 2	2.4 28	32	2	9	77	87
767	01-15-85	o	0	0.0	0.3	42	<0.1	;	,	} ;		} '	\		} ;	} ;	}	} ;	;	1
494	02-21-85	54	17.0	0.0	0.1	204	¢0.1	100	100	007	_	_			_					
797	03~11~85	87	20.9	2.0	9.0	220	<0.1	86	86	86	86	86	. 92		*	86 86	86 8	86	86	86
1310/A1.0.	01-15-85	0	0	0.0	0.0	36	(0.)	ł	;	;		, }	;		1	;	1	ì	,	
1310/AL.U.	02-20-85	54	18.3	2.5	0.0	229	60.1	100	001	100					•					1
1310/41.0.	03-06-85	84	18.1	6.1	0.0	198	<0.1	001	100	8	001	901	90	90	2001	01 001	100 100		8	8
1881	01-15-85	0	9	1.0	1.8	52	60. 1	;	;	:			· :		;	}	;	ł	1	1
1961	02-14-85	57	21.1	3.8	1.7	115	¢0.1	86	86	98										
1881	02-04-85	87	20.8	2.0	4.5	150	<0.1	66	66	66	66	66	6	6	\$	66	66 66	8	8	66
1381/A1.0.		0	0	0.0	0.8	•	<0.1	1	}	ļ	;	;	' !	;	}	1	}	1	1	1
1 181/41.0.	02-12-85	54	16.9	2.0	0.5	191	<0.1	101	101	101			-					•	1	1
1381/41.0.	02-26-85	87	20.2	10.3	1.3	175	<0.1	66	66	66	66		. 66	66	5	66 66	66 6			5

Al.U. Fuel percolated through alumina

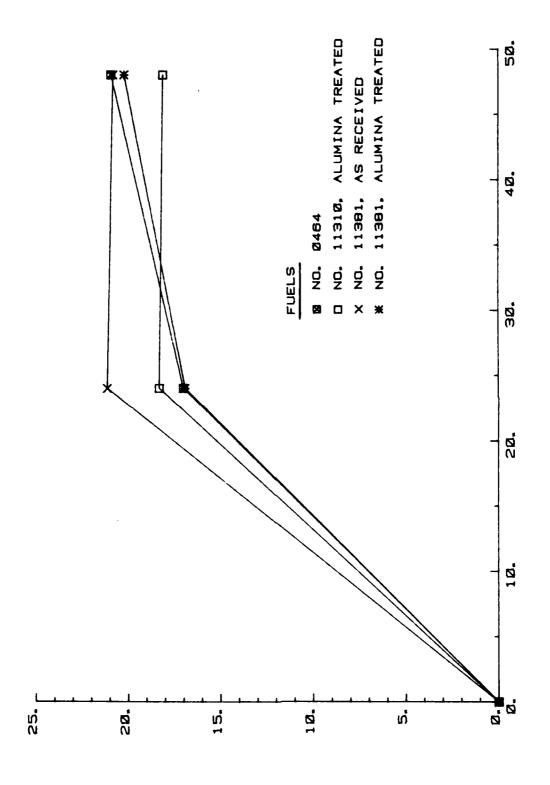
TABLE 4. OXIDATION OF KEROSENES AT 180°C UNDER AN INITIAL OXYGEN PRESSURE OF 100 PSIG

		Time.	Oxygen Used.	Peroxides.	S.J.Gum,	Water.	TAN.					Oxyge	п Вер.	i et 1o	1 (ps:) Va	Time	(hous	rs)		
Fuel No.	Date	hr	mole/L	ppm	mg/100mL	ppm	mg KOtt/g		2	3	4	6	8	10	12	14	16	18	20	22	24
464		0	0	0	0.2	42	<0.1							_	_	_	_		_	_	
464	10-31-84	6	17.6	2.8	0.8	56	-	108	108	108	108	108									-
164	10-15-84	24	18.0	4.5	0.5		-	110	110	110	110	110	110	110	110	110	110	110	110	110	110
310/AL.O.		0	0	0	0.6	29	<0.1	_					_				-		_	_	_
310/A1.O.	11-12-84	4	25.4	44	0.5	28	<0.1	108	108	107	107		_		-	-	_		-	_	
1310/AL.O.	10-23-84	6	23.0	60	0.5	40		110	015	110	110	109	-			_				-	
310/A1.0.	01-24-85	16	24.4	263	2.5	342	_	115	115	115	115	114	113	111	109	107	105		_	-	_
310/A1.0.	10-17-84	24	99.3	949	2.7	450		111	111	111	111	109	107	103	98	93	88	83	77	71	65
381		0	0	0	2.6	40	<0.1			-		-	_	_	_	-	-		_	-	-
381	01-11-85	6	9.3	49	8.5	112	_	116	116	116	116	116				_				_	_
381	01-31-85	6	11.7	34	2.3	132	<0.1	112	112	112	112	112			_		_		_	_	_
381	Average	6	10.5	42	5.4	122	<0.1														
381	St. Dev.	0	1.7	11	4.4	14															
381	01-08-85	10	20.4	146	10.9	257		111	111	111	111	110	110	108	_	_	_		_	_	_
1381	01-10-65	10	19.9	96	7.4	197	0.2			113				108	_	_		_	_	_	_
-	Average	10	20.2	121	9, 2	227	0.2			•••	•••		•••			-		_	_	_	
	St. Dev.	0	0.4	35	2.5	42															
381	11-28-84	16	57.0	648	3.8	148	0.1	107	107	107	107	107		105	102 105	97 98	88 89		_		_
381	01-26-85	16	52.8	851	26.6	421 285	0.2	112	112	112	112	112	111	103	103	70	07		_	_	_
381	Average	16	54.9	750	15.2																
1381	St. Dev.	0	3.0	144	16.1	193	0.1														
381	10-02-84	24	137.0	1900	56.0			109	109	109	109	109	108	106	103	96	91	81	66	56	4
361	10-05-84	24	128.6	2028	50.5			108	108	108	106	106	107	106	103	99	93	84	73	60 46	4.7
1381	11-26-84	24	151.7	1941	70.0	936	0.6	109	109	109	109	109	108	106	103 102	97	87 84	75 71	60 57	41	34 31
1381	01-15-65	24	153.8 142.8	2397 2067	58.8	486 711	0.6	111	111	111	111	111	109	100	102	74	-	/1	31	41	٠.
1381 1381	Average St. Dev.	24	12.1	227	10.1	318															
1361	01-16-85	48	236.6	2080	598	3295	5.3	(110)	(105)	(102)	(99)	(83)	(73)	(65)	(56)	(49)	(43)	(37)	(32)	(27)	(23
381/41 6			0	0	1.0	37	<0.1					_			_						
381/A1.0. 381/A1.0.	11-07-84	0	26.7	91	1.1	37	<0.1	106	106	106	106	_	_	_	_	_	_	_	_	_	_
						-		-													
381/AL.O.	10-2 9-8 4	6	29.7	209	0.7			106	106	107	106	104		_	-	_	-	-	_	_	
381/A1.0.	12-06-84	6	25.4	145	1.1	15	<0.1	108	108	107	107	106			_		_		-	_	_
381/A1.0.	12-17-84	6	26.7	120	0.5	23	<0.1	108	108	108	107	106		_	-		_	_	-		_
381/A1.0.	Average	6	27.3	158	0.8 0.3	19 6	<0.1 —														
381/A1.0.	St. Dev.	0	2.2	46	0.3	•	_														
381/A1.0.	11-30-84	10	46.5	114	0.2	29	<0.1	108	108	108	108	108	106	107		_	_	_		~-	-
38'/AL.O.	12-18-84	10*	35.7	387	2.2	39	<0.1	107	107	107	107	106	103	99	-	-		_			-
381/AL.O.	Average	10	41.1	251	1.2	34	<0.1														
381/A1.O.	St. Dev.	0	7.6	193	1.4	7															
381/A1.O.	12-19-84	16*	96.9	1400	13.5	509	0.2	107	107	107	106	104	101	96	89	79	68		_	_	_
381/A1.0.	01-03-65	160	115.8	1479	32.1	835	0.4	107	107	106	105	102	97	89	79	67	55	-	_	_	_
	01-30-85	16*	77.5	1381	15.7	631	-	112	112	112	112	112	111	109	105	98	89	_	_		_
381/A1.0.	PRETOVA	16	96.7	1420	20.4	658	0.3														
	~~~	•	19.2	52	10.2	165	0.1														
381/A1.0.	St. Dev.	0																			
381/AL.O. 381/AL.O.		24	153.1	1613	76.3		_	112	112	112	111	110	106	102	96	87	77	66	54	43	3 :
381/AL.O. 381/AL.O. 381/AL.O.	St. Dev.		153.1 166.8	1613 1749	76.3 70.7	_	_	112	112		111	110	106	102	96 93	87 83	77 72	66 60	54 48	43 36	
1381/A1.0. 1381/A1.0. 1381/A1.0. 1381/A1.0. 1381/A1.0.	St. Dev.	24							110	110		109									24
1381/AL.O. 1381/AL.O. 1381/AL.O. 1381/AL.O.	5t. Dev. 10-08-84 10-10-84	24 24	166.8	1749	70.7			111	110	110	110	109	106	100	93	83	72	60	48	36	31 24 35

Al.O. Fuel percolated thru alumina

* Second batch of fuel percolated through activated nameral alumin.

^( ) System representized after first 24 hours of oxidation. Presents readings taken during the second 24 hours of stress



STRESS DURATION, HOURS

MMOLE

OXYGEN CONSUMPTION,

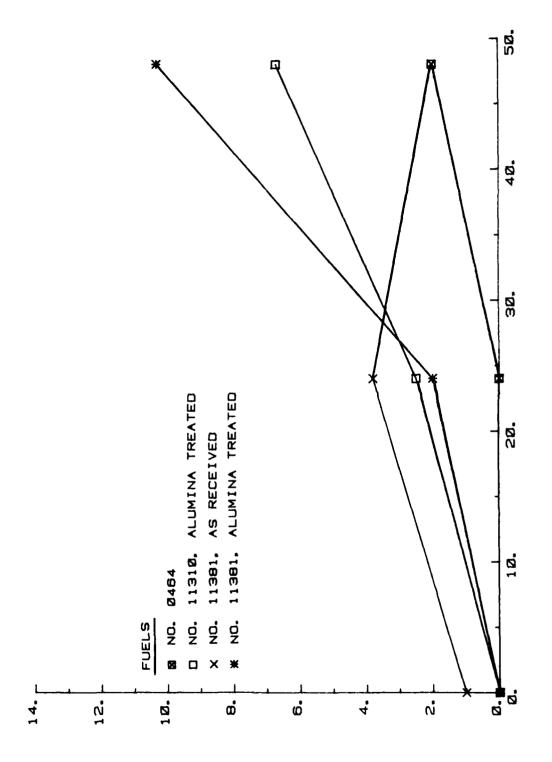
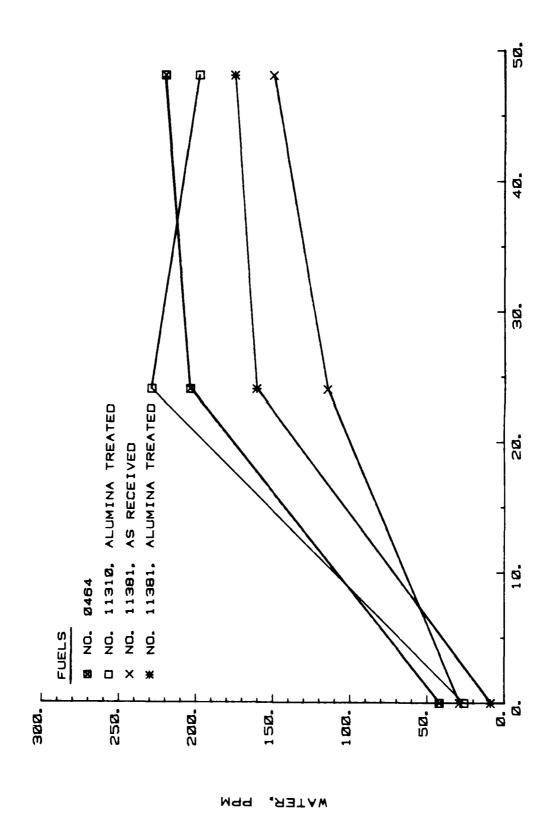


FIGURE 7. OXIDATION AT 60°C WITH 100 PSI OXYGEN (PEROXIDES, PPM)

STRESS DURATION, HOURS

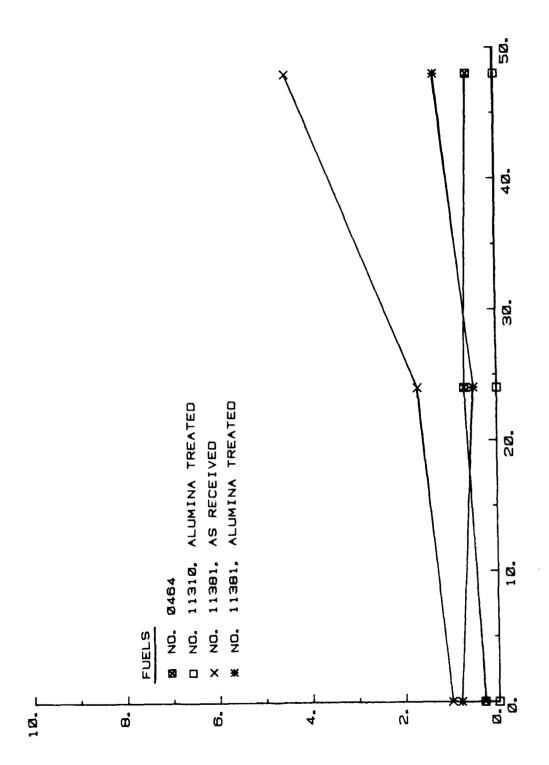
Mdd

PEROXIDES.



PIGURE 8. OXIDATION AT 60°C WITH 100 PSI OXYGEN (WATER, PPM)

STRESS DURATION, HOURS

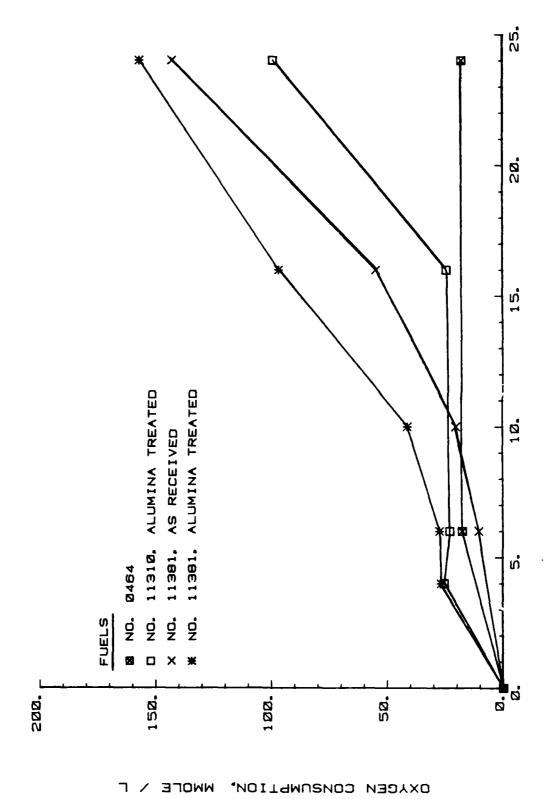


STRESS DURATION, HOURS

FIGURE 9. OXIDATION AT 60°C WITH 100 PSI OXYGEN (GUM, MG/100 ML)

ØØ 1

COM¹



STRESS TIME, HOURS

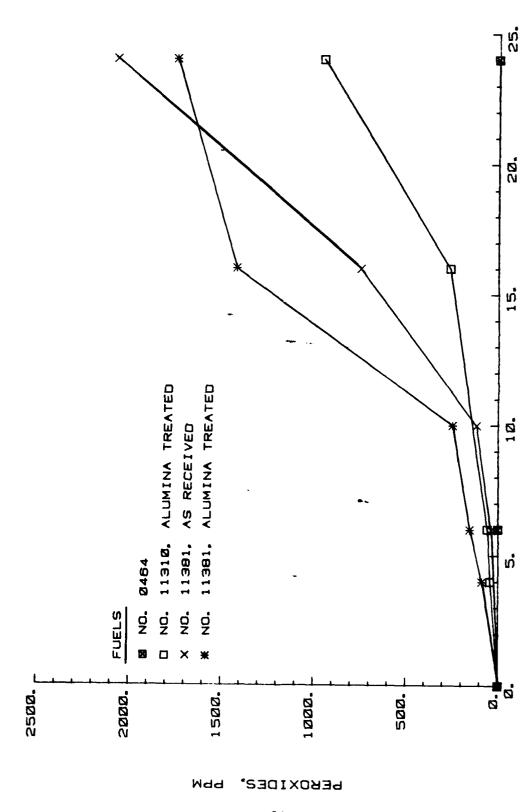


FIGURE 11. OXIDATION AT 100°C WITH 100 PSI OXYGEN (PEROXIDES, PPM)

STRESS TIME, HOURS

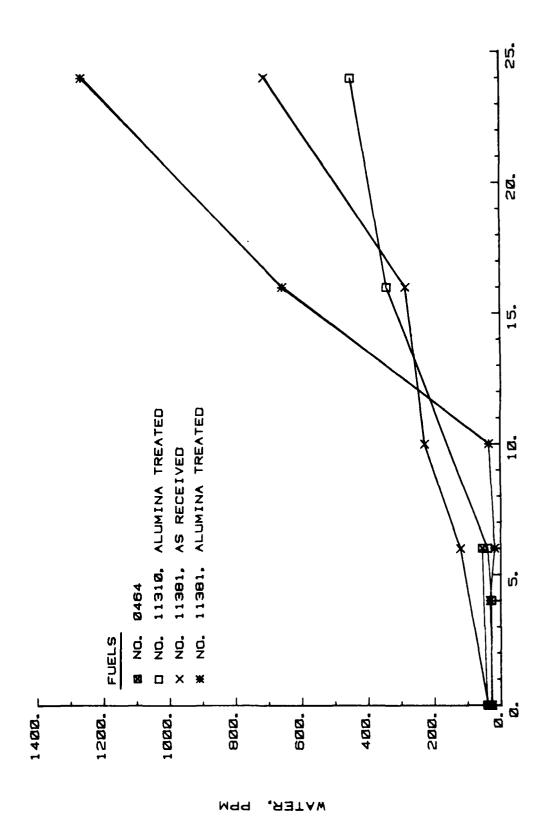
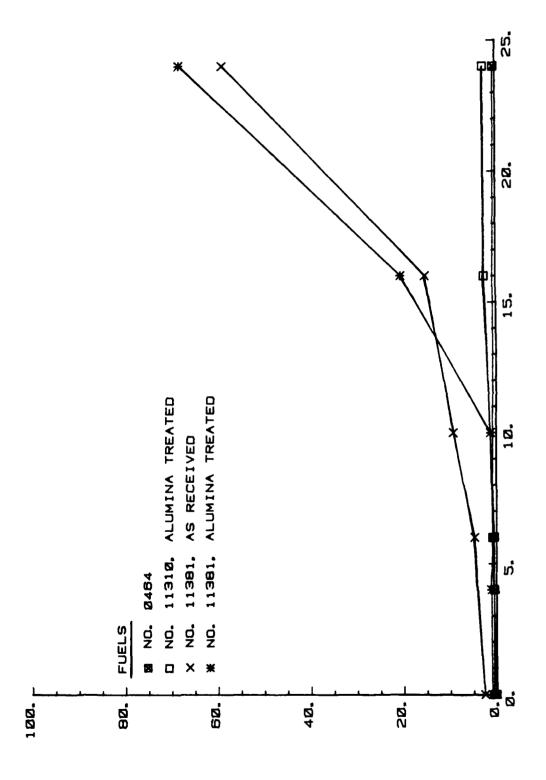


FIGURE 12. OXIDATION AT 100°C WITH 100 PSI OXYGEN (WATER, PPM)

STRESS TIME, HOURS



PIGURE 13. OXIDATION AT 100°C WITH 100 PSI OXYGEN (GUM, MG/100 ML)

SCHOOL CONTROL

STRESS TIME, HOURS

001

water and gum that were generated, gave inconclusive results. In no instance, were measurable amounts of acidic components produced.

Oxidation of the same kerosenes at 100°C under the influence of 689 kPa (100 psig) of oxygen (99.99%) produced substantial degradation while producing only small amounts of acidic components. When the results of these experiments were compared with those of the bottle storage, it was apparent that both methods agree on the identity of the most stable fuel (No. 0464) and the least stable fuel (No. 11381, alumina-treated). However, the two intermediate fuels' positions have been interchanged. No explanation for this observation may be offered at this time.

The tentative relative ranking of the four fuels after ultimate indicated stress periods by the three test procedures are summarized in Table 7.

A further observation may also be made from the pressure gauge readings during test, as given under the heading of "Oxygen Depletion" in Table 4 and 5. As discussed previously, the reactor was pressurized to a predetermined extent, e.g., 100 psig, at a measured room temperature. Upon completion of the test, the reactor and its contents were cooled to room temperature, and pressure and temperature readings were taken again. From these data, the total amount of consumed oxygen was calculated. During the test, a continuous recording was made of the reactor pressures. As the reactor was heated from room temperature, e.g., 23° to 100°C, the pressure inside the reactor should have increased from 689 kPa (100 psig) to 896 kPa (130 psig). It was found, however, that a maximum pressure of 800 kPa (116 psig) developed 1 hour after the reactor reached the set temperature of 100°C. The average initial pressure was 758 kPa (110 psig) with a standard deviation of 17.2 kPa (2.5 psig). Similarly, at 60°C the calculated oxygen pressure of 786 kPa (114 psig) was higher than the observed pressures given in Table 4. In both cases, the balance of the oxygen was assumed to be the increased amount of oxygen that dissolved in the fuel due to the increase in temperature.

TABLE 7. RANKING OF FUELS ACCORDING TO VARIOUS TESTS

# Fuel Identification Number

Property	"Stable Fuel"			"Unstable Fuel"	
Bottle Storage: 43°C/24 week	s				
Oxygen in vapor phase	0464		11381	11310A	11381 A
Oxygen in liquid phase	0464		11381	11310A	11381 A
Peroxide content	0464		11381	11310A	11381A
Water content	0464	~	11381	11310A	11381 A
Gum content	0464	~	11310A	11381	11381 A
Reactor: 60°C/100 PSIG/48 ho	ours				
Oxygen consumed	0464	$\simeq$	11381	11381 A	11310A
Peroxide content	0464	~	11381	11310A	11381A
Water content	11381		11381 A	11310	0464
Gum content	11310A		0464	11381 A	11381
Reactor: 100°C/100 PSIG/24 h	nours				
Oxygen consumed	0464		11310A	11381	11381A
Peroxide content	0464		11310A	11381 A	11381
Water content			11310A	11381	11381A
Gum content	0464	~	11310A	11381	11381A

### V. CONCLUSIONS AND RECOMMENDATIONS

Through the generally accepted 43°C (110°F) bottle storage method of accelerated fuel aging, the relative ratings of four selected fuels' oxidative tendencies were established. In this method, storage for about 12 weeks produced results comparable to a full year's storage under ambient conditions. To develop a practical test method for the prediction of peroxide potential of fuels, experimental conditions were sought that the oxidative tendencies of fuels could be assessed within a reasonable time, i.e., less than 48 hours. Accordingly, a matrix of experiments was designed to allow selection of those reaction conditions that would give results comparable to the 43°C bottle storage experiments. Additionally, the results of these experiments would allow the development of global reaction kinetics to aid the determination of fuel peroxidation potential. From the partially completed experimental matrix (as defined earlier), fuel stressing was completed at 60° and 100°C under an initial oxygen pressure of 689 kPa (100 psig). Experimental results at 60°C gave inconclusive results. Results of the 100°C experiments produced essentially self-consistent results that also agreed with those of the bottle storage for the most stable and least stable fuels. Results of the two intermediate stability fuels, however, were interchanged.

It is recommended that the proposed experimental matrix be completed to allow completion of project objectives. However, as a consequence of inconclusive findings at 60°C and 689 kPa (100 psig), it is recommended that the original matrix be modified to delete the 345 kPa (50 psig) condition at 60°C. This work should then be followed by determination of reliability and accuracy of this possible new practical analytical method.

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- 2. D.A. Netzel and P.M. Hunter, "Hydrocarbon-Type Analysis of Jet Fuels by ¹H and ¹³C NMR," DOE/LETC/RI-81-1, May 1981.
- 3. Proposed ASTM Test Method for Distillate Fuel Storage Stability at 43°C.

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CDR DAVID TAYLOR NAVAL SHIP R&D CTR ATTN: CODE 2830 (MR BOSMAJIAN) CODE 2759 (MR STRUCKO) CODE 2831	1 1 1	CDR NAVAL FACILITIES ENGR CTR ATTN: CODE 1202B (MR R BURRIS) 200 STOVWALL ST ALEXANDRIA VA 22322	i
CG FLEET MARINE FORCE ATLANTIC ATTN: G4 (COL ROMMANTZ) NORFOLK VA 23511	ı	CDR NAVAL AIR ENGR CENTER ATTN: CODE 92727 LAKEHURST NJ 08733	i
CDR NAVAL SHIP ENGINEERING CENTER ATTN: CODE 6764 (MR. BOYLE) PHILADELPHIA PA 19112	1	COMMANDING GENERAL US MARINE CORPS DEVELOPMENT & EDUCATION COMMAND ATTN: DO74 (LTC WOODHEAD) QUANTICO VA 22134	l
JOINT OIL ANALYSIS PROGRAM - TECHNICAL SUPPORT CTR BLDG 780 NAVAL AIR STATION PENSACOLA FL 32508	ı	OFFICE OF CHIEF OF NAVAL RESEARCH ATTN: ONT-07E (MR ZIEM) ARLINGTON, VA 22217	1
PROJ MGR, M60 TANK DEVELOPMENT ATTN: USMC-LNO US ARMY TANK-AUTOMOTIVE	ı	CHIEF OF NAVAL OPERATIONS ATTN: OP 413 WASHINGTON DC 20350	1
COMMAND (TACOM) WARREN MI 48397  DEPARTMENT OF THE NAVY HQ, US MARINE CORPS		FLEET MARINE FORCE PACIFIC ATTN: G4 (COL HARMS) CAMP H.M. SMITH HI 96861	1
ATTN: LPP (MAJ WALLER)  LMM/3 (MAJ WESTERN)  WASHINGTON DC 20380	1	CDR NAVY PETROLEUM OFC ATTN: CODE 43 CAMERON STATION	1
CDR NAVAL AIR SYSTEMS CMD ATTN: CODE 53645 (MR MEARNS) WASHINGTON DC 20361	1	DEPARTMENT OF THE AIR FORCE	
CDR NAVAL AIR DEVELOPMENT CTR ATTN: CODE 60612 WARMINSTER PA 18974	1	HQ, USAF ATTN: LEYSF (COL CUSTER) WASHINGTON DC 20330	1
CDR NAVAL RESEARCH LABORATORY ATTN: CODE 6170 CODE 6180 CODE 6110 (DR HARVEY) WASHINGTON DC 20375	1 1 1	HQ AIR FORCE SYSTEMS CMD ATTN: AFSC/DLF (MAJ VONEDA) ANDREWS AFB MD 20334	1

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US AIR FORCE WRIGHT AERONAUTICALAB ATTN: AFWAL/POSF (MR CHURCHILL) AFWAL/POSL (MR JONES) AFWAL/MLSE (MR MORRIS) WRIGHT-PATTERSON AFB OH 45433		SPACE ADMINISTRATION VEHICLE SYSTEMS AND ALTERNATE FUELS PROJECT OFFICE ATTN: MR CLARK LEWIS RESEARCH CENTER CLEVELAND OH 44135	1
CDR SAN ANTONIO AIR LOGISTICS CTR ATTN: SAALC/SFT (MR MAKRIS) SAALC/MMPRR KELLY AIR FORCE BASE TX 78241	1 1	DEPARTMENT OF TRANSPORTATION FEDERAL AVIATION ADMINISTRATION ATTN: AWS-110 800 INDEPENDENCE AVE, SW WASHINGTON DC 20590	1
CDR WARNER ROBINS AIR LOGISTIC CTR ATTN: WRALC/MMTV (MR GRAHAM) ROBINS AFB GA 31098	1	US DEPARTMENT OF ENERGY CE-1312 ATTN: MR ECKLUND FORRESTAL BLDG. 1000 INDEPENDENCE AVE, SW WASHINGTON DC 20585	1
CDR USAF 3902 TRANSPORTATION SQUADRON ATTN: LGTVP (MR VAUGHN) OFFUTT AIR FORCE BASE NE 68113	1	ENVIRONMENTAL PROTECTION AGENCY AIR POLLUTION CONTROL 2565 PLYMOUTH ROAD ANN ARBOR MI 48105	1
CDR HQ 3RD USAF ATTN: LGSF (MR PINZOLA) APO NEW YORK 09127	I	AGENCY FOR INTERNATIONAL DEVELOPMENT ATTN: MR D HOOKER M/SER/EOMS/OPM, ROOM 2155A11 WASHINGTON DC 20523	1
CDR DET 29 ATTN: SA-ALC/SFM CAMERON STATION ALEXANDRIA VA 22314	1		

**OTHER GOVERNMENT AGENCIES** 

MR. GROBMAN)

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